

3. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites and fungi as well as to improve its physical-mechanical properties, according to claims 1 and 2, CHARACTERIZED because the impregnation is performed with wood whose humidity content fluctuates between 1 and 50%, and preferably less than 30 %.
4. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 3, CHARACTERIZED because the soluble silicate used is sodium silicate, in a silice solution with a concentration between 1 and 28 % in weight, preferably between 4 and 16% in weight.
5. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 4, CHARACTERIZED because the soluble silicate used is potassium silicate, in a silicon solution with a silicon dioxide concentration between 1 and 28 % in weight, preferably between 4 and 16% in weight.
6. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 5, CHARACTERIZED because the impregnation is performed under vacuum and pressure conditions in one or more successive cycles of vacuum and pressure.
7. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 6, CHARACTERIZED because the impregnation is performed under a pressure of 1 to 20 atmospheres, preferably 8 to 15 atmospheres during a period of 10 to 300 minutes, preferably from 15 to 60 minutes.
8. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 5, CHARACTERIZED because the impregnation stage is performed by immersion at atmospheric pressure.
9. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 5, CHARACTERIZED because the impregnation stage is performed with showers or other aspersion methods.
10. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 9, CHARACTERIZED because to the solution of an alkaline hydroxide and a silicate, used for impregnation, soluble metaborate salts are added.
11. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 10, CHARACTERIZED because the metaborate is produced previously through the reaction of boric acid with a highly dissociated hydroxide and is later added to the silicate solution.
12. Accelerated petrification process of lignocellulose materials to increas its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 10, CHARACTERIZED because the metaborate is

produced through the reaction of soluble sodium or potassium tetraborate with a highly dissociated hydroxide and it is later added to the silicate solution.

13. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 12, CHARACTERIZED because the concentration of the impregnating solution has a content of soluble metaborate of 0.02 to 0.7% of boron in weight, preferably between 0.1 and 0.3 % of boron in weight.
14. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 13, CHARACTERIZED because the final concentration of boron in the lignocellulose material is 0.08 and 3.20 kg/m<sup>3</sup>, preferably 0.40 to 1.40 kg/m<sup>3</sup> of wood and of 4 to 126 kg/m<sup>3</sup> of silice, preferably between 18 and 74 kg/m<sup>3</sup> of wood.
15. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 14, CHARACTERIZED because the insolubilization of silice and boron can be facilitated in the interior of the lignocellulose material due to a subsequent washing stage with water, with water acidulated with inorganic acids, organic acids, and/or salts or a mixture of them.
16. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 15, CHARACTERIZED because the washing is performed with a solution that contains small quantities of sulfuric acid, hydrochloric acid, nitric acid, boric acid, phosphoric acid, acetic acid, formic acid or a mixture of them.
17. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 16, CHARACTERIZED because the washing is performed with a soluble alkaline earth element solution (calcium, strontium or barium, as soluble chlorides or nitrates).
18. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 17, CHARACTERIZED because the washing with water or with a liquid of more acidic characteristics than those of the impregnating liquid occurs under vacuum and pressure conditions.
19. Accelerated petrification process of lignocellulose materials to increase its characteristics of resistance to fire, termites, and fungi as well as to improve its physical-mechanical properties, according to claims 1 to 17, CHARACTERIZED because the washing with water or with a liquid of more acidic characteristics than those of the impregnating liquid occurs via baths, or other methods of immersion, or showers or other methods of aspersion.

## SUMMARY

A fast ¿(Accelerated)? petrification process for lignocellulose materials, and especially for low density wood for construction, housing, industrial, decorative, agricultural as well as other uses like: the fabrication of paneling, windows, doors, floors, posts, beams, poles, furniture, terraces, bridges, machine parts and many others for interior and exterior use, and even in contact with soil and water, is produced through an impregnation with an alkaline hydroxide solution and a water soluble silicate, alternatively including soluble salts of a metaborate, and its insolubilization in situ due to the action of acidic organic groups liberated by the components of the lignocellulose material due to the conditions characteristic of the process and the presence of carbon dioxide in the surrounding air.

The petrified lignocellulose material inflames and putrefies with great difficulty and is not attacked by termites or other insects; its aspect changes slightly over time, increasing its hardness and improving its characteristics of mechanical resistance and dimensional stability. The properties can be adjusted according to the intended final use by varying the concentration of the constituents and the operating process conditions.

## LITERATURE

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## EXPERIMENTAL EXAMPLES

### Example 1

Ten wood samples of radiata pine of 50x20x15 mm with a water content of 12% were treated in an array of impregnators at a laboratory scale. The operating conditions used in the impregnation were the following:

Item	Operating Conditions
No. of samples	10
Liquor concentration (K/A) (%)	7/0.05
Initial Vacuum (min)	10
Impregnation at 14 (KG/cm <sup>2</sup> ) (min)	30
Intermediate Vacuum (min)	10
Water washing (min)	15
Final Vacuum (min)	10

The termites were placed in glass recipients that contained the test sample in humid sand, sterilized and rich in silicates. Subsequently, the recipients were placed in a culture chamber at temperatures of  $24.5 \pm 1^\circ\text{C}$  and a relative humidity of  $75 \pm 5\%$ . The tests were performed according to the European Norm EN 117/ACI of 1990 to determine the efficacy threshold with respect to *Reticulitermes sp* (subterranean termites) under laboratory conditions. The duration of the test was 8 weeks and was performed in triplicate.

At the end of the test, the test samples were removed from the recipients and a visual inspection was performed to characterize the termite attack according to its location, extension and depth. A census of termites was also performed to determine the survival and/or mortality rate of the population.

The following table was used as a base for the visual evaluation of the test pieces.

Attack level	Wood Characteristics
0	No attack
1	Tentative attack: Cleanliness or surface rubbing of a immeasurable depth or an attack exactly at a depth of 0.5 mm if this is restricted to three areas of less than 3 mm of diameter each one.
2	Light attack: surface attack (<1 mm) and limited in extension to at most 1/10 of the surface of the test piece or one orifice of depth less than 3 mm.
3	Medium attack: surface attack (<1 mm) of more than 1/10 of the surface of the test piece or with clear-cut holes that do not extend into caverns and with a depth greater than 3 mm.
4	Strong attack: Surface erosion that involves more than 1/10 of the surface of the test piece or penetrating attack that is greater than 3 mm and extends into caverns in the test mass and could lead to a very advance state of destruction.

#### Results

Test Pieces	Attack Level
<u>Untreated wood</u>	
Sample 1	Strong Attack (Level 4)
Sample 2	Strong Attack (Level 4)
Sample 3	Strong Attack (Level 4)
<u>Treated wood</u>	
Sample 1	No attack (Level 0)
Sample 2	Tentative Attack (Level 1)
Sample 3	Tentative Attack (Level 1)

According to the Table, the treated wood presented a high resistance to termite attack, differing from the untreated the wood that manifested a strong attack.

#### Example 2

Wood samples of radiata pine of 50x20x15 mm with a water content of 12% were treated in an array of impregnators at a laboratory scale. The operating conditions used in the impregnation were the following:

Item	Operating Conditions
No. of samples	10
Liquor concentration (K/A) (%)	5/0.2
Initial Vacuum (min)	10
Impregnation at 14 (KG/cm2) (min)	30
Intermediate Vacuum (min)	10
Water washing (min)	15
Final Vacuum (min)	10

The treated wood, like the untreated wood samples, experienced termite action following the same procedure described in Example 1.

## Results

Test Pieces	Attack Level
<u>Untreated wood</u>	
Sample 1	Strong Attack (Level 4)
Sample 2	Strong Attack (Level 4)
Sample 3	Strong Attack (Level 4)
<u>Treated wood</u>	
Sample 1	No attack (Level 0)
Sample 2	No attack (Level 0)
Sample 3	Tentative Attack (Level 1)

It was verified that the treated wood is resistant to termite attack, which was not the case of the untreated wood that presented a strong attack.

### Example 3

Wood samples of radiata pine of 50x25x15 mm with a water content of 12% were treated in a array of impregnators at a laboratory scale. The operating conditions used in the impregnation were the following:

Item	Operating Conditions
No. of samples	10
Liquor concentration (K/A) (%)	3/0.2
Initial Vacuum (min)	10
Impregnation at 14 (KG/cm <sup>2</sup> ) (min)	30
Intermediate Vacuum (min)	10
Water washing (min)	15
Final Vacuum (min)	10

The treated wood, like the untreated wood samples, experienced termite action following the same procedure described in Example 1.

## Results

Test Pieces	Attack Level
<u>Untreated wood</u>	
Sample 1	Strong Attack (Level 4)
Sample 2	Strong Attack (Level 4)
Sample 3	Strong Attack (Level 4)
<u>Treated wood</u>	
Sample 1	No attack (Level 0)
Sample 2	Tentative Attack (Level 1)
Sample 3	Tentative Attack (Level 1)

Once again, it was established that the treated wood is resistant to termite action, while the untreated wood was strongly attacked.

### Example 4

Three wood samples of radiata pine of 15cm x 15cm x 10 mm thickness with a water content of 12% were treated in an array of impregnators at a laboratory scale. The operating conditions used in the impregnation were the following:

Liquors		Wood	
K (%)	A (%)	K (%)	A (%)
9.0	0.22	7.0	0.3

The tests pieces were evaluated with respect to their fire resistance following the Norm ISO 5660-1 "Fire test – reaction to fire – rate of heat release from building products", known as the calorimetric cone methodology.

#### Results

Measured Parameters	Average of the Results for the three samples	
	Untreated Wood	Treated Wood
Thickness (mm)	12.3	11.0
Ignition Time (s)	13.0	18.3
Rate of heat release (KW/m <sup>2</sup> )	88.1	40.76
Effective Combustion Heat (MJ/kg)	13.8	16.43
Reason of weight loss (m <sup>2</sup> /kg)	0.05	0.02
Smoke release (m2/kg)	88.8	48.59
Prod. of Carbon Monoxide (kgCO/kg)	0.027	0.05
Prod. of Carbon Dioxide (kgCO <sub>2</sub> /kg)	1.83	2.68

According to the table, the treated wood is superior in all the evaluated characteristics with respect to untreated wood.

#### Example 5

Three wood samples of radiata pine of 15cm x 15cm x 10 mm thickness with a water content of 12% were treated in an array of impregnators of laboratory scale. The operating conditions used in the impregnation were the following:

Liquors		Wood	
K (%)	A (%)	K (%)	A (%)
12.0	0.1	9.0	0.1

The treated and untreated samples were submitted to fire resistance tests similar to those signaled in Example 4.

#### Results

Measured Parameters	Average of the Results for the three samples	
	Untreated Wood	Treated Wood
Thickness (mm)	12.3	13.5
Ignition Time (s)	13.0	22.0
Rate of heat release (KW/m <sup>2</sup> )	88.1	50.46
Effective Combustion Heat (MJ/kg)	13.8	10.2
Reason of weight loss (m <sup>2</sup> /kg)	0.05	0.043
Smoke release (m2/kg)	88.8	50.3
Prod. of Carbon Monoxide (kgCO/kg)	0.027	0.031
Prod. of Carbon Dioxide (kgCO <sub>2</sub> /kg)	1.83	1.56

It was observed that the treatment is effective in improving the wood's fire resistance.

#### Example 6

Three wood samples of radiata pine of 15cm x 15cm x 10 mm thickness, dried to a water content of 12% were treated in a array of impregnators of laboratory scale. The operating conditions used in the impregnation were the following:

Liquors		Wood	
K (%)	A (%)	K (%)	A (%)
11.0	0.6	10.0	0.6

Fire resistance tests similar to those described in Example 4 were performed on the treated and untreated samples.

#### Results

Measured Parameters	Average of the Results for the three samples	
	Untreated Wood	Treated Wood
Thickness (mm)	12.3	13.5
Ignition Time (s)	13.0	22.0
Rate of heat release (KW/m <sup>2</sup> )	88.1	50.46
Effective Combustion Heat (MJ/kg)	13.8	10.2
Smoke release (m <sup>2</sup> /kg)	0.05	0.043
Reason of weight loss (m <sup>2</sup> /kg)	88.8	50.3
Prod. of Carbon Monoxide (kgCO/kg)	0.027	0.031
Prod. of Carbon Dioxide (kgCO <sub>2</sub> /kg)	1.83	1.56

Under the described conditions, the petrification process was found to be very effective in providing the wood with fire resistance qualities.

#### Example 7

Three wood samples of radiata pine of 15cm x 15cm x 10 mm thickness, dried to a water content of 12% were treated in an array of impregnators at a laboratory scale. The operating conditions used in the impregnation were the following:

Liquors		Wood	
K (%)	A (%)	K (%)	A (%)
5.0	0.2	5.9	0.25

Fire resistance tests similar to those described in Example 4 were performed on the treated and untreated samples.

#### Results

Measured Parameters	Average of the Results for the three samples	
	Untreated Wood	Treated Wood
Thickness (mm)	12.3	11.5
Ignition Time (s)	13.0	22.0
Rate of heat release (KW/m <sup>2</sup> )	88.1	60.8
Effective Combustion Heat (MJ/kg)	13.8	10.3
Reason of weight loss (m <sup>2</sup> /kg)	0.05	0.051
Smoke release (m <sup>2</sup> /kg)	88.8	54.0
Prod. of Carbon Monoxide (kgCO/kg)	0.027	0.024
Prod. of Carbon Dioxide (kgCO <sub>2</sub> /kg)	1.83	1.54

The treated wood exhibited better fire resistance properties than the untreated wood.

#### Example 8

Three wood samples of radiata pine of 15cm x 15cm x 10 mm thickness, dried to a water content of 12% were treated in an array of impregnators at a laboratory scale.

The operating conditions used in the impregnation were the following:

Liquors		Wood	
K (%)	A (%)	K (%)	A (%)
3.0	0.2	3.2	0.22

#### Results

Fire resistance tests similar to those described in Example 4 were performed on the treated and untreated samples.

#### Results

Measured Parameters	Average of the Results for the three samples	
	Untreated Wood	Treated Wood
Thickness (mm)	12.3	11.0
Ignition Time (s)	13.0	22.0
Rate of heat release (KW/m <sup>2</sup> )	88.1	83
Effective Combustion Heat (MJ/kg)	13.8	14
Reason of weight loss (m <sup>2</sup> /kg)	0.05	0.05
Smoke release	88.8	41.0
Prod. of Carbon Monoxide (kgCO/kg)	0.027	0.025
Prod. of Carbon Dioxide (kgCO <sub>2</sub> /kg)	1.83	1.5

Once again, an improvement in fire resistance was observed in the wood treated by the petrification process with respect to the untreated wood.